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(57) Abstract

The use of P_2O_5 and/or B_2O_3 as a component to improve the refractoriness of inorganic fibres comprising SiO₂, and CaO and/or MgO is described. The inorganic fibres have a composition such that SiO₂ + P_2O_5 -(58 + (if MgO > 10, 0.5 x (MgO-10) else 0)) > -2.4 wt.%.

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SALINE SOLUBLE INORGANIC FIBRES

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This invention relates to saline soluble inorganic fibres.

Saline soluble inorganic fibres have been described in several patent specifications, see for example WO93/15028. Fibres are required to be soluble in saline solution so that inhaled or ingested fibres dissolve rather than providing a source of irritation or otherwise affecting health. WO93/15028 showed that fibres comprising SiO₂, CaO and MgO and having a silica content of greater than 58% (or greater than 58% plus 0.5 times (wt%MgO - 10) if MgO > 10wt%) had suitable shrinkage characteristics at 800°C and 1000°C to be usable as refractory materials. A further feature of WO93/15028 was the use of the percentage of non-bridging oxygens present to predict the solubility of fibres in physiological saline solution.

Various subsequent applications have described the effect of P_2O_5 and B_2O_3 on solubility - see for example WO95/29135. P_2O_5 is alleged to have a solubilising effect on such fibres.

The German government have proposed a fibre classification which turns on a variable K_I which is defined as:

 $K_I = \Sigma$ (Na,K,B,Ca,Mg,Ba -oxide) - 2* Al-oxide (the amounts of the oxides being expressed as weight %)

According to the proposed fibre classification if K_I is greater than 40 the fibre requires no health warnings. If K_I lies between 30 and 40 the fibre requires health warnings to be made. If K_I is less than 30 more serious marking is required (it is labelled as a carcinogen). It is readily apparent that it is difficult to provide a high K_I fibre ($K_I > 40$) while still providing a refractory fibre like that of WO93/15028 (SiO₂>58wt%), there being a very narrow window of compositions to meet.

As a result of investigating fibre compositions that may meet the fibre classification and yet still be refractory enough to meet the standard of WO93/15028 (shrinkage of less than 3.5% at both 800°C and 1000°C) the applicants have found that addition of P_2O_5 to compositions allows a broader range of refractory fibres to be produced than had previously been appreciated. They have also found that B_2O_3 , previously thought to be

extremely detrimental to refractoriness, has a similar, although lesser, effect and that both P_2O_5 and B_2O_3 may be used in the fibres of WO93/15028.

The applicants have found that the refractoriness of the P_2O_5 and B_2O_3 containing fibres of the present invention is dependent on the sum of the amounts of SiO_2 and P_2O_5 (expressed in wt%)

It appears that a further factor that may be important in determining the refractoriness of a fibre is the percentage of non-bridging oxygens. If this percentage is 61.4% or more (calculated on the basis of the amounts of the components SiO₂, CaO, MgO, P₂O₅, and B₂O₃) the fibres tend to fail shrinkage tests at 800°C and 1000°C (failure being defined as a shrinkage of 3.5% or more).

Accordingly the present invention provides the use of P_2O_5 and/or B_2O_3 as a component to improve the refractoriness of inorganic fibres comprising SiO_2 , and CaO and/or MgO, the inorganic fibres having a composition such that

$$SiO_2 + P_2O_5 - (58 + (if MgO > 10, 0.5 \times (MgO - 10) else 0)) > -2.4wt%$$

The invention provides further such fibres in which the percentage of non-bridging oxygens is less than 61.4%.

Further features of the invention are apparent from the claims in the light of the following description.

The percentage of non-bridging oxygens (%N.B.O.) is calculated by converting the weight percentages of SiO₂, CaO, MgO, P₂O₅, and B₂O₃ to molar amounts and inserting these amounts into the equation:-

%N.B.O. =
$$\frac{2*(CaO + MgO + P_2O_5 + B_2O_3)}{(2*SiO_2 + CaO + MgO + 5 \times P_2O_5 + 3 \times B_2O_3)} \times 100$$

The reason the amounts of CaO, MgO, P_2O_5 , and B_2O_3 are doubled in the numerator to this equation is that each contributes two non-bridging oxygens. The reason terms are multiplied in the denominator to this equation is to reflect the number of oxygen atoms each molecular formula possesses.

Table I shows the results of a first set of shrinkage and solubility tests on compositions comprising SiO₂, CaO, MgO, P₂O₅, and B₂O₃ as main

ingredients. In this table the analysed compositions are normalised to 100%. It is clear from these compositions that where the percentage of non-bridging oxygens calculated on the basis of the amounts of the above named components is greater than 61.4% (those fibres lying above line A of Table I) the fibres fail the shrinkage tests, having shrinkages of greater than 3.5% at either or both of 800°C and 1000°C.

WO93/15028 stressed the importance of alumina content and the fibres lying between lines B and A of Table I show that alumina contents of greater than lwt% are damaging to the shrinkage properties of fibres.

The applicants have also found that the combined amount of CaO and MgO is important. Those fibres lying between lines C and B have a combined CaO and MgO content of greater than 42wt% and also fail the shrinkage tests:

The fibres below line C have a percentage of non-bridging oxygens less than 61.4%, an alumina content of less than 1wt%, and a combined CaO and MgO content of less than 42wt%. All of these fibres pass the shrinkage tests. These fibres fall within the compositional ranges:-

SiO ₂	52.4 - 57.85wt%
CaO	22.2 - 39.4wt%
MgO	1.96 - 17.4wt%
P_2O_5	0.82 - 7.8wt%
B_2O_3	0 - 1.95wt%
Al ₂ O ₃	<1wt%

The solubility results presented in Table I were obtained by the methods described in WO93/15028 and show a high solubility for all of the fibres produced.

It can be seen that all of the fibres below line C have a K_I of more than 35 and more than half have a K_I of more than 40.

Further testing resulted in the data presented in Table II. The data presented are as in table I but an additional column entitled deviation shows the result of looking to the difference between the sum of the SiO₂ and P₂O₅ contents and the SiO₂ amount predicted to be needed by WO93/15028 for a fibre to be refractory (shrinkage of less than 3.5% at both 800°C and 1000°C. The figure given is found by calculating the sum

$$SiO_2 + P_2O_3 - (58 + (if MgO > 10, 0.5 \times (MgO - 10) else 0))$$

If this is less than -2.4wt% the fibres fail. The fibres that failed are shown in plain text, those that passed in bold text, and those that were difficult to form in italics.

More than 12.5wt% P₂O₅ is undesirable as it causes difficulties in making the fibres.

While the above description and the claims refer to P_2O_5 , B_2O_3 , SiO_2 , CaO and MgO it will be clear to the person skilled in the art that the pure materials need not be used and that provision of these components in combined form (e.g. provision of P_2O_5 in the form of mixed oxide phosphates) is part of the invention.

30			đ	Chemical C		emposition (XRF		- Weight percent)	Ę			Z	Shrinker	ş		Solubility (ppm)					S S S S S S S S S S S S S S S S S S S
LTP	0"3	OBJN	202	SiO2	A1203	Na2O	K20	B203	Fc203	2002	SrC		800°C 1000°C	သူ	9	Og W	SiO	B203	Total	CaO+MgO	
LTP 8	24.95	19.18	3.41	\$1.69	0.25	0.30	0.05		0.17	< 0.05	< 0.05	44.0	40.0	40.0	8	86	177		328	4.14	68.5%
LTP 9	24.81	18.66	5.10	50.42	0.38	0.31	€0.0		0.17	0.13	< 0.05	43.0	23.9	38.8	\$	115	193		367	43.47	68.1%
LTP11	25.13	19.07	2.51	52.54	0.28	0.23	0.03		0.17	< 0.05	< 0.03	43.9	\$	39.1	25	94	174		323	44.20	68.0%
LTP16	_	12.27	3.39	\$1.59	0.26	0.42	90.0		0.17	< 0.03	< 0.05	4	49.1		79	92	300		355	#.=	66.1%
1.TP10	_	17.89	2.48	54.46	0.21	0.28	0.03		91.0	< 0.03	< 0.03	42.3	3.62	1.61	85	8	169		317	42.37	64.7%
LTP 4		17.78	3.31	53.85	0.31	0.26	0.03		0.15	0.25	< 0.05	41.5	3.71	4.77	26	98	180		331	41.83	64.3%
LTP 5	24.22	17.17	4.91	52.72	0.33	0.30	< 0.05		0.14	0.21	< 0.05	41.0	3.63	5.39	65	106	161		362	41.40	<u>\$</u>
LTP17	_	5.54	3.41	51.22	0.40	0.42	0.0		91.0	0.38	< 0.05	43.6	45.2	43.8	83	32	161		306	43.94	63.9%
1.1723		5.56	2.57	52.23	0.34	9.46	0.07		0.15	< 0.03	< 0.05	4	42.90		22	52	188		310	44.18	63.7%
LTP14		10	4.90	51.96	0.30	0.45	0.03		0.15	0.23	< 0.05	£.8	3.24	3.92	26	69	161		338	41.95	63.0%
TPI		27.95	3.26	57.2	< 0.03	0.13	< 0.05		0.17	< 0.05	< 0.05	39.4	5.72	5.26	8	1117	188		335	39.23	63.0%
TP12		11.35	3.36	53.52	0.32	0.31	90.0		0.15	< 0.05	< 0.05	42.0	2.55	30.1	22	72	207		361	42.27	63.6%
1.TP20		11.35	2.52	54.14	0.32	0.31	90.0		91.0	0.10	< 0.05	42.1	3.38	29.7	88	71	200		356	42.40	62.6%
1.TP15	_	5.70	\$ 05	51.22	0.31	0.43	0.10		91.0	0.13	< 0.05	42.5	3.41	5.03	88	35	204		327	42.59	62.2%
I TP 3	_	16.69	6.70	\$2.58	0.25	0.29	< 0.05		0.14	97.0	< 0.05	39.4	23.3	29.5	43	991	14		350	39.58	61.9%
1.TP 7	10.37	27.85	3.29	58.18	< 0.03	0.15	< 0.03		91.0	< 0.05	< 0.05	38.4	6.01	15.5	36	132	. 152		320	38.23	61.4%
1 TP52	- -		4 89	2 8 2	38	0,38	80	ê. 20.03	<u> </u>	<u>6</u> 0.0	<u>6</u> 0.0	32.6	32.1	-	72	74	140		386	36.40	\$6.0%
L.1P51			1.62		1.38	0.29	0.02	<0.05	0.26	<0.05	<0.05	37.3	3.07	19.6	82	69	139		310	39.70	58.4%
1.TP29	13	38	1.23	55.09	0.0	0.39	613		0.19	0.17	\$0.05	0. \$	45.9	-	9/	01	206		191	42.38	58.8%
1771		5.58	2.54	54.19		0.46	0.07		0.15	< 0.05	< 0.05	42.0		35.5	88	34	208		38	42.20	60.3%
1.TP30	-	1.96	2.22	<u>\$</u>	\$	3	3		2	<u>6</u> 0.0	< 0.05	1 0	1.74	2.04	72	П	209		192	41.36	57.5%
LTP41		9.48	0.83	55.63		0.30	0.07	88.	91.0	<0.05	< 0.05	42.5	1.20	132	87	9	194	20	361	40.84	%0.09
LTP6		10.45	3.34	\$5.65		0.32	0.03		0.15	< 0.05	< 0.05	40.3	1.89	2.76	\$	22	172		289	40.28	\$9.0%
LTP34		9.81	1.68	57.3	0.25	0.31	0.07		0.13	<0.03	< 0.05	1.0	3 .	1.79	76	51	881		315	40.25	\$8.0%
LTP43	_	99.6	1.68	\$6.19	0.28	0.32	0.07	Ξ	0.13	<0.05	< 0.05	=	0.97	1.84	62	8	187	12	327	40.19	58.8%
LTP42	_	9.56	98.0	57.13	0.27	0.33	0.07	1.08	0.15	<0.05	< 0.05	=	1.04	<u>8</u> :	2	જ	192	12	344	40.13	58.2%
LTP47					15.0	0.31	0.03	0.03	0.1	<0.05	<0.05	39.3	1.97	2.14	88	104	197		359	39.60	%0.19
LTP38				•	0.31	0.30	80.0	0.94	0.15	<0.05	< 0.05	40.3	1.07	9.	=	22	175	0	292	39.56	55.4%
LTP2			4.87	54.25		0.24	< 0.05		0.16	0.58	< 0.03	38.8	2.24	3.05	53	96	167	-	316	39.45	80.8%
1.TP39	_	4.73	1.67	57.39	0.27	0.30	80.0	8	0.14	<0.05	< 0.05	0.0	1.47	1.93	33	33	20	9	284	39.08	55.2%
LTP 1	23.29	15.66	333	10.72	0.24	0.22	90.0		0.14	< 0.05	0.0	38.7	<u></u>	1.71	63	68	175		327	38.94	58.7%
LTP48	32	6.87	7.8	52.4	0.52	0.34	0.03	<0.05	0.13	0.18	<0.05	38.2	1.24	1.53	*	***	202		337	38.87	57.7%
LTP40	33.67	4.75	0.86	57.85	0.38	0.31	0.08	1.95	0.15	<0.05	< 0.03	40.0	2.1	2.39	\$	32	194	23	<u>5</u>	38.42	54.5%
LTP26	33.69	4.56	3.7	56.93	0.36	0.43	90.0		0.1	0.07	< 0.05	38.0	1.22	94.	6	78	193		312	38.25	54.0%
1.TP27		9.33	3.66	57.32	0.22	0.36	0.03		0.14	<0.05	< 0.05	38.2	0.99	1.16	63	84	173		188	38.24	55.5%
1.TP46	28.4	8.69	2.67	59	0.79	0.33	90.0	<0.05	0.13	<0.05	<0.05	36.9	16:0	0.99	7	\$	173		292	37.09	53.3%

TABLE II (Part 1)

17.00	5 0.08 9.17 11 0.08 9.17 12 0.05 13 0.05 14 0.05 16 0.07 17 0.08 18 0.05 18 0.05 19 0.07 10 0.08		0,000000	50.54 50.54 50.54 50.54 50.00 57.20 57.20 57.24 57.20 57.24 57.20 57.24 57.20 57.24 57.20	MgO 7203 S102 19.18 3.41 \$1.69 19.07 2.51 \$2.54 6.74 \$0.54 \$0.64 25.18 \$0.6 \$4.00 27.29 3.26 \$7.20 27.85 3.29 \$8.18 17.89 2.48 \$4.46 17.78 3.31 \$3.85 17.17 4.91 \$2.22 10.47 12.93 41.37 10.40 \$4.50 \$4.50 \$5.4 \$1.27 \$4.50 \$6.4 \$4.7 \$4.02 \$6.4 \$4.7 \$4.00 \$6.5 \$1.27 \$4.50 \$6.5 \$6.5 \$6.25 \$6.5 \$6.5 \$6.25 \$6.5 \$6.2 \$7.52 \$7.5 \$7.52 \$7.5 \$7.52	8 3.41 51.69 50.54 50.54 50.6 54.00 3.26 54.00 3.26 54.00 2.52 57.24 2.52 57.24 3.129 58.18 3.31 53.85 4.91 52.72 12.93 41.37 12.93 41.37 12.93 41.37 12.93 41.37 12.93 41.37 12.93 41.37 12.93 41.37 12.93 51.59 12.93 51.59 12.93 51.59
53 98 177 328 44.14 55 94 174 323 44.20 59 115 193 129 463 39.09 59 115 193 367 43.47 40.35 39.23 40.35 30 117 188 335 39.23 40 36 119 210 39.53 56 95 180 317 42.37 65 106 191 362 44.11 67 200 30 367 41.40 83 20 191 367 41.40 82 20 199 367 41.40 82 29 199 310 44.18 73 73 255 21 42.4 38.58 71 108 83 208 37.48 5 82 69 159 310 39.73 6	0.10 0.11 0.10 0.10 0.10 0.10 0.10 0.10	55 0.08 11 0.08 12 0.05 13 0.05 14 0.05 16 0.07 17 0.08 18 0.05 18	9 0.25 0.30 0.05 1 0.28 0.25 0.08 2 - 0.38 0.31 0.35 0.19 0.35 0.19 0.35 0.19 0.26 0.05 0.36 0.31 0.08 0.36 0.31 0.08 0.36 0.31 0.08 0.37 0.40 0.42 0.40 0.42 0.07 0.72 0.55 0.33 0.36	1 \$1.69 0.25 0.30 0.05 1 \$2.54 0.28 0.25 0.08 50.54 0.57 0.40 0.08 \$6.42 0.38 0.31 0.25 \$7.20 0.19 0.25 0.05 \$7.24 0.38 0.19 0.13 \$7.24 0.35 0.19 0.15 \$8.18 0.21 0.28 0.05 \$8.18 0.21 0.26 0.05 \$1.59 0.26 0.42 0.06 \$1.59 0.26 0.42 0.05 \$41.37 2.31 0.36 0.05 \$41.37 2.31 0.42 0.07 \$40.02 0.72 0.42 0.07 \$40.02 0.72 0.26 0.07 \$35.24 0.23 0.26 0.07	19.18 3.41 \$1.69 0.25 0.30 0.05 19.07 2.51 \$2.54 0.28 0.25 0.05 6.74 \$0.54 0.57 0.40 0.08 18.66 \$1.0 \$0.42 0.31 0.03 25.18 \$0.6 \$4.00 0.19 0.25 27.95 \$1.26 \$7.20 0.13 0.13 27.85 \$1.25 \$7.24 0.35 0.19 17.89 \$2.48 \$4.46 0.21 0.28 0.05 17.78 \$3.31 \$5.85 0.31 0.26 0.05 17.71 4.91 \$2.72 0.33 0.30 0.05 10.40 \$2.49 41.37 \$2.31 0.56 0.05 10.40 \$2.40 0.04 0.08 0.05 0.05 10.40 \$2.40 0.04 0.00 0.04 0.00 \$2.4 \$2.4 0.02 0.05 0.05 0.05	24.95 19.18 3.41 51.69 0.25 0.30 0.05 25.13 19.07 2.51 \$25.84 0.28 0.25 0.06 24.81 18.66 \$1.0 \$0.54 0.57 0.40 0.08 15.17 25.18 \$0.66 \$4.00 0.19 0.25 0.01 11.28 27.95 3.26 \$4.00 0.19 0.23 0.13 11.28 27.95 3.26 \$4.00 0.19 0.23 0.19 10.37 27.81 3.29 \$8.18 0.19 0.15 0.05 24.48 17.89 2.48 \$4.46 0.21 0.28 0.05 24.04 17.78 3.31 \$3.85 0.31 0.26 0.05 24.21 17.71 4.91 \$2.72 0.33 0.30 0.05 32.13 10.47 12.93 41.37 2.31 0.05 0.05 34.38 2.54 3.41 \$1.27
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42 41 179 262 42.60 79 58 200 30 367 41.40 83 32 191 306 43.94 82 29 199 313 44.18 73 73 255 21 424 38.58 17 108 83 208 37.48 72 74 140 286 36.45 82 69 159 310 39.73 88 33 204 36.75 42.59	. 4 - 4 - 2 m 2 m 2 m 3 m 2 m 2 m 2 m	0.08 3.01 0	2.31 0.56 0.05 0 0.36 0.31 0.08 3.19 0 0.40 0.42 0.07 0 0 0.72 0.55 0 0 0 1 0.34 0.46 0.07 0 0 0.25 0 0 0	41.37 2.31 0.56 0.05 0 54.50 0.36 0.31 0.08 3.19 0 1 51.22 0.40 0.42 0.07 0 0 40.02 0.72 0.55 0 0 0 0 35.23 0.34 0.46 0.07 0 0 0 0 35.24 0.26 0.26 0 0 0 0 0 0 0 0	10.47 12.93 41.37 2.31 0.56 0.05 0.05 0.05 0.05 0.05 0.05 0.05 0.05 0.05 0.05 0.07	10.47 12.93 41.37 2.31 0.56 0.05 3.19 0 10.40 54.50 0.36 0.31 0.08 3.19 0 9 5.54 3.41 51.22 0.40 0.42 0.07 0 9.46 14.72 40.02 0.72 0.55 0 0 0 2.56 2.57 52.23 0.34 0.46 0.07 0 0 9.55 19.83 35.24 0.24 0.18 0.08 3.01 0 14.20 3.752 0.44 0.18 0.08 3.01 0
79 58 200 30 367 41.40 83 32 191 306 43.94 60 57 196 313 43.84 73 25 199 310 44.18 73 73 255 21 424 38.58 17 108 83 208 37.48 72 74 140 286 36.45 82 69 159 310 39.73 88 35 204 327 42.59	A - A - A 20 00 00 01 01 20 00 4	2 0.07 2 0 6 0.07 0 6 0.08 3.01 0	0.36 0.31 0.08 3.19 0 0.40 0.42 0.07 0.72 0.55 0.07 0.34 0.46 0.07	54.50 0.36 0.31 0.08 3.19 0 1 51.22 0.40 0.42 0.07 0 <td< td=""><td>10.40 54.50 0.36 0.31 0.08 3.19 0 5.54 3.41 51.22 0.40 0.42 0.07 9.46 14.72 40.02 0.72 0.55 0 5.56 2.57 52.23 0.34 0.46 0.07 9.55 19.83 35.24 0.23 0.26 0 14.20 37.52 0.44 0.18 0.08 3.01 0</td><td>10.40 54.50 0.36 0.31 0.08 3.19 0 9 5.54 3.41 51.22 0.40 0.42 0.07 19 0 9.46 14.72 40.02 0.72 0.55 0 0 0 0 0 0 0 2.56 2.57 52.23 0.34 0.46 0.07 0</td></td<>	10.40 54.50 0.36 0.31 0.08 3.19 0 5.54 3.41 51.22 0.40 0.42 0.07 9.46 14.72 40.02 0.72 0.55 0 5.56 2.57 52.23 0.34 0.46 0.07 9.55 19.83 35.24 0.23 0.26 0 14.20 37.52 0.44 0.18 0.08 3.01 0	10.40 54.50 0.36 0.31 0.08 3.19 0 9 5.54 3.41 51.22 0.40 0.42 0.07 19 0 9.46 14.72 40.02 0.72 0.55 0 0 0 0 0 0 0 2.56 2.57 52.23 0.34 0.46 0.07 0
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73 73 255 21 424 38.58 17 108 83 208 37.48 72 74 140 286 36.45 82 69 159 310 39.73 88 35 204 327 42.59		0.08 3.01	0.23 0.26	35.24 0.23 0.26	19.83 35.24 0.23 0.26 57.52 0.44 0.18 0.08 3.01	9.55 19.83 35.24 0.23 0.26 14.20 57.52 0.44 0.18 0.08 3.01
75 73 255 21 424 38.58 17 108 83 208 37.48 72 74 140 286 36.45 82 69 159 310 39.73 88 35 204 327 42.59	a 5 3 5 2 2	0.08 3.01	_		57.52 0.44 0.18 0.08 3.01	14.20 57.52 0.44 0.18 0.08 3.01
17 108 83 208 37.48 38.13 42.32 42.32 42.32 72 74 140 286 36.45 82 69 159 310 39.73 88 35 204 327 42.59	א מיועיוש		0.44 0.18 0.08 3.01	0.44 0.18 0.08 3.01		
72 74 140 286 36.45 82 69 159 310 39.73 88 35 204 327 42.59			0.27 0.12	59.45 0.27 0.12	0.27 0.12	2.53 59.45 0.27 0.12
72 74 140 286 36.45 82 69 159 310 39.73 88 35 204 327 42.59	2 2 2	Abo	Abo	Abo	Abo	Abo
72 74 140 286 36.45 82 69 159 310 39.73 88 33 204 327 42.59	2 2		0.07 0.44	16.68 0.07 0.44	0.07 0.44	14.55 46.68 0.07 0.44
72 74 140 286 36.45 82 69 159 310 39.73 88 35 204 327 42.59	2	0.05	0.09 0.70 0.05	42.67 0.09 0.70 0.05	0.09 0.70 0.05	14.02 42.67 0.09 0.70 0.05
72 74 140 286 36.45 82 69 159 310 39.73 88 35 204 327 42.59		54 0.05 0.14	0.69 0.54 0.05	44.91 0.69 0.54 0.05	0.69 0.54 0.05	12.64 44.91 0.69 0.54 0.05
1.02 72 74 140 286 36.45 -0.24 82 69 159 310 39.73 wr%- -1.72 88 35 204 327 42.59	Š	Abo	Abo	Abor	Abo	Abo
-0.24 82 69 159 310 39.73 wr?«	1.38	0.05	2.06 0.28 0.05	54.88 2.06 0.28 0.05	2.06 0.28 0.05	4.90 54.88 2.06 0.28 0.05
-1.72 88 35 204 327 42.59		29 0.07 0.26	1.38 0.29 0.07	56.65 1.38 0.29 0.07	1.38 0.29 0.07	1.62 56.65 1.38 0.29 0.07
42.50 3.41 5.03 -1.72 88 35 204 327 42.59	<:					
	0.16	01.0	0.31 0.43 0.10	0.43 0.10	51.22 0.31 0.43 0.10	5.70 5.05 51.22 0.31 0.43 0.10
69	0.15	0.05	0.30 0.45 0.05	0.45 0.05	51.96 0.30 0.45 0.05	4.90 51.96 0.30 0.45 0.05
42.90 2.62 2.78 -1.65 57 42 223 322 42.70 67.0%	0.19	0.05	0.19 0.53 0.05	44.34 0.19 0.53 0.05	0.19 0.53 0.05	12.01 44.34 0.19 0.53 0.05
40.88 1.72 1.95 -1.56 71 54 203 318 42.05 65.1%	0.17	0.05	0.84 0.46 0.05	46.79 0.84 0.46 0.05	0.84 0.46 0.05	9.65 46.79 0.84 0.46 0.05
0.05 39.45 0.01 0.00 1.84 71 89 222 376 39.18 60.1%	0.15	0.05	0.17 0.56 0.05	50.26 0.17 0.56 0.05	0.17 0.56 0.05	9.58 50.26 0.17 0.56 0.05
Above here SiO2 content less than \$2mt%						

TABLE II (Part 2)

ğ			ō	Chemical Composition (XRF	ompostt	ion (XR		- Welght percent)	Œ		-	3	8	Shrinkage	2		Solubility (ppm)	(bpm) k				%N.B.O.
1.TP	Og ၁	MgO	P205	SiO2	A1203	N=20	K20	B203	Fe203	202	တ္ဌ	Ц.	6	ပ 000	1000°C Deviation	ဒ္ဓ	Og W	SiO2	B203	Total	CaO+MgO	
1193	22.89	16.69	6.70	\$2.58	0.23	0.29			0.14	0.46	T	10.37	23.30	05.62	10.7	7	166	E		330	39.38	L
L.TP20	31.05	11.35	2.52	54.14	0.32	0.31	90.0		0.16	0.10	_	42.13	3.38	29.70	-2.01	82	17	200		356	42.40	
LTP 2	23.35	16.10	4.87	54.25	6.	0.24			0.16	0.58		38.77	1.24	3.03	-1.93	8	8	167		316	39.45	%8:09
I.TP12	30.93	11.35	3.36	\$3.52	0.32	0.31	90.0		0.15			42.00	2.55	30.10	-1.79	E	12	207		361		62.6%
1.TP21	36.62	5.58	2.54	\$4.19	0.39	8	0.07		0.15			41.95	•	35.50	-1.27	88	34	208		300	42.20	%6.09
L.TP48	31.90	6.85	7.78	52.24	0.52	0.34	0.05		0.15	0.18		38.10	1.24	1.53	2.02	3	89	205		337	38.75	\$1.7%
				_					Abov	Above here SiO2 content 52wt% to	O2 coate	ont 52wt	% to less	less than 55 wr%	wt%							
LTP47	22.30	17.48	8,4	55.45	0.31	0.31	0.05		0.10		 	39.52	1.97	2.14	-2.29	88		197		359	39.78	61.0%
1,TP64	20.81	18.41	2.52	\$7.63	0.22	0.26			0.14			39.04	3.01	3.73	-2.05	4	76	197		319	39.22	59.7%
LTP68	20.08	18.77	4.55	55.92	0.30	0.24	_		0.14			38.49	3.8	4.16	-1.92	25	88	226		366	38.85	60.2%
LTP29	40.29	2.09	1.23	\$5.09	0.43	0.39	0.12		0.19	0.17		42.03	45.85	•	-1.68	76	01	206		292	42.38	58.8%
1.TP41	31.36	87.6	0.85	55.63	0.27	0.30	0.07	 88.	0.16			42.55	1.20	131	1.52	82	3	194	20	361	40.84	60.0%
L.TP71	38.31	0.65		56.51	0.55	0.20	0.09	3.54	0.14			41.69	0.59	1.43	-1.49	23	~	278	35	408	38.96	\$6.78
LTP30	39.40	1.8	1.12	55.25	0.45	0.4	0.10		0.21			\$3.	1.74	2.8	-0.S3	77	=======================================	508		192	41.36	\$7.5%
LTP 1	23.29	15.66	3,33	57.01	0.24	0.22	0.0		0.14		0.05	38.74	=	1.71	-0.49	3	86	175		327	38.2	58.7%
L.TP43	30.51	9.68	1.68	56.19	0.28	0.32	0.0	1.1	0.15			41.13	0.97	ヌ	-0.12	62	8	187	12	327	40.19	58.8%
LTP37	35.40	4.77		\$7.92	0.3	0.31	60.0	1.05	0.15			40.99	1.57	2.13	-0.08	37	30	195	13	275	40.16	\$6.1%
LTP32	30.01	8.5		57.95	0.32	0.23	0.0	2.69	0.18			40.92	89:	2.83	6.03	26	\$	3	77	334	38.54	56.3%
L.TP73	36.93	0.62		57.96	6.49	6.23	60.0	3.5	0.13			40.43	27	3.00	3.	76	7	264	2	382	37.55	52.6%
L.TP42	30.55	9.56	98.	57.13	0.27	0.33	0.0	8.	0.15			41.06	3	18.	-0.07	75	65	192	12	344	40.12	58.1%
LTP38	34.82	4.73	0.82	57.84	0.31	0.30	80.0	0.94	0.15		-	40.26	1.07	3 :	0.66	3	32	175	6	292	39.56	55.4%
LTP40	33.67	4.75	98.0	57.85	0.38	0.31	98.0	<u>x</u>	0.15			40.00	1.15	239	0.71	\$	32	194	23	291	38.42	54.5%
1.TP 6	29.83	10.45	3.34	\$5.65	0.21	ĩ	0.05		0.15			40.23	<u>&</u>	2.76	0.76	જ	25	172		189	40.28	29.0%
1.TP69	19.17	17.56	39.	57.93	0.31	ព្រ			0.13			36.34	1.23	1.68	0.81	\$	28	241		378	36.73	\$6.5%
L.TP34	30.44	18.6	83:	57.30	0.25	2	0.07	-	0.15		_	40.13	8.	1.79	0.98	76	25	88		315	40.25	\$8.0%
LTP39	34.35	4.73	1.67	57.39	0.27	వై	80.0	30.	0.14			39.98	1.47	1.93	1.06	32	33	203	91	28-	39.08	55.1%
L.TP26	33.69	4.56	3.73	\$6.95	0.36	9.5	9.6		0.14	0.0		38.02	1.22	07:	1.68	2	38	193		312	38.25	%0.Z
1.7727	28.91	9.33	3.66	57.32	0.22	0.36	0.03		0.14			38.21	0.99	1.16	2.99	67	48	173		288	38.24	55.5%
									Above	here Sit)2 conte	at SSwr	Above here SiO2 content 55wt% to less than 58wt%	then 58v	wr%							

TABLE II (Part 3)

Г	_	-	S	_	*	.0		•		_	•	\•		_	•	_	
002%			701 15		37.6	55.09		75.57	55.19		3.	\$ 6.13	. C)		51.3%	51.3%	
		CENTINGO	36.81		38.10	36.27	37 40	27.40	37.50	30 10	20.17	39.23	14 01		300	37.25	
	1	7	283	*	***	325	223	775	387	200		2	35	;	<u>c+</u> 5	392	
	8203	3				23	00	•	=	34	3	2	32	:	2		
Solubility (ppm)	ξ	2	169	100		193	170		Š	8		3	193	9	100	175	
Sebber	Ç ₀ M	1	ಪ	87		8	32		8	3		7	7	- 53	3	\$	
	Ş		8	4		\$	90		2	37	8	8	8	-	5	7	
3	000°C Deviation		2	79.07		9.0	09.0		2.0	8.0	5	3	1.8.1	1 78	2	3.93	
Shrinkage		Ī	3.19	2.37		9.70	3.85	*	4.86	3.12	3.74		2.71	3.15		0.99	or more
	၁.008	1	7.65	2.28		3.37	1.65	316	3.13	3:	3.16	?	3.	2.17		0.91	58Wf%
2			3	37.72		2	40.40	90.07	€7:2	40.13	40.03		38.82	35.76		3.50	conten
	So		_			_							0.19		_		Above here SiO2 content 58wt% or more
	202															7	Above
Cept	Fe203	3	£.53	0.13	71.0	<u>.</u>	0.15	0 10		0.15	0.17		0.13	0.15	;	5.5	
- weight percent)	B203				3,74	2.43	3.09	20	3	2.10	0.96		3.16	<u>1</u> .8			
	K20				8	3	0.08	0.00	3	80.0	90.08		- - - -	90.0	20.0	3	
	Ne.20	36.0	7.5	0.22	0 27		0.31	0 29		8	0.28	ì	ا ا	0.24	0.23	3	
	SiO2 AI2O3	0.34	***	0.30	0 27	-	0.28	0.28		0.27	0.27	3.0	S S	3.0	000	29.5	
		K0 17	į	\$8.75	80 64		28.60	58.70		<u>R</u>	59.01	100	39.81	62.48	40 26		ĺ
3	P205	4 29	2	2.50											3 68 69 78	3	
	Q Z	31 16		17.74	13.60		4.76	9.20		70.	9.03	8	8	11.40	K 73		
		15.65		20.36	22 67		32.72	28.30	,	33.3/	30.20	30.05	3.6	24.10	LTP46 28.52		
5	e E	1.TPAK		LIP65	LTP72		C173	LTP31		Š.	L.TP33	Trous		LTP4S	TP46		

CLAIMS

1. The use of P₂O₅ or B₂O₃ as a component to improve the refractoriness of inorganic fibres comprising SiO₂, and CaO and/or MgO, to produce inorganic fibres having a composition having a shrinkage of less than 3.5% when exposed to 1000°C for 24 hours and having a shrinkage of less than 3.5% when exposed to 800°C for 24 hours, the fibres having a composition such that

$$SiO_2 + P_2O_5 - (58 + (if MgO > 10, 0.5 \times (MgO - 10) else 0)) > -2.4wt%$$

- 2. The use of P₂O₅ or B₂O₃ as a component to improve the refractoriness of inorganic fibres as claimed in claim 1 in which the percentage of non-bridging oxygens is less than 61.4%.
- 3. The use of P₂O₅ or B₂O₃ as a component to improve the refractoriness of inorganic fibres as claimed in claim 1 or claim 2 in which the fibres fall within the compositional range:-

SiO ₂	44 or more
CaO	20 - 40wt%
MgO	0 - 18wt%
P_2O_5	0- 12.5wt%
B_2O_3	0 - 4wt%

4. The use of P₂O₅ or B₂O₃ as a component to improve the refractoriness of inorganic fibres as claimed in claim 3 in which the fibres fall within the compositional range:-

$$SiO_2 \qquad \qquad 52 - <58 wt\% \ [52 - <58 + 0.5'(MgO-10)wt\% \ if \\ \qquad \qquad MgO > 10 wt\%]$$
 CaO
$$22 - 40 wt\%$$
 MgO
$$0 - 17.5 wt\%$$
 MgO + CaO
$$<42 wt\%$$

$$P_2O_5 \qquad 0.5 - 10 wt\%$$

$$B_2O_3 \qquad 0 - 2 wt\%$$

5. The use of P₂O₅ or B₂O₃ as a component to improve the refractoriness of inorganic fibres as claimed in claim 3 in which the fibres fall within the compositional range:-

CaO	20.36 - 39.4wt%
MgO	0.62 - 21.16wt%
P_2O_5	0 - 12.01wt%
B_2O_3	0 - 3.54 wt%

6. Saline soluble inorganic fibres having a shrinkage of less than 3.5% when exposed to 1000°C for 24 hours and having a shrinkage of less than 3.5% when exposed to 800°C for 24 hours, in which:-

$$SiO_2 + P_2O_5 - (58 + (if MgO > 10, 0.5 \times (MgO - 10) else 0)) > -2.4wt%$$

7. Saline soluble inorganic fibres as claimed in claim 6 comprising:

SiO ₂	44 or more
CaO	20 - 40wt%
MgO	0 - 18wt%
P_2O_5	0-12.5wt%
B_2O_3	0 - 4wt%

8. Saline soluble inorganic fibres as claimed in claim 7 comprising:-

 $\begin{tabular}{lllll} MgO > 10wt\% \\ CaO & 22 - 40wt\% \\ MgO & 0 - 17.5wt\% \\ MgO + CaO & < 42wt\% \\ P_2O_5 & 0.5 - 10wt\% \\ \end{tabular}$

SiO₂

B₂O₃

and in which the percentage of non-bridging oxygens calculated on the basis of the amounts of the above named components is less than 61.4%.

52 - <58wt% [52 - <58+0.5'(MgO-10)wt% if

9. Saline soluble inorganic fibres as claimed in claim 7 comprising:-

0 - 2wt%

 $\begin{array}{lll} SiO_2 & 44.34 - 62.48 \\ CaO & 20.36 - 39.4wt\% \\ MgO & 0.62 - 21.16wt\% \\ P_2O_5 & 0 - 12.01wt\% \\ B_2O_3 & 0 - 3.54wt\% \end{array}$

10. Saline soluble inorganic fibres as claimed in claim 6 in which the fibres have a composition:-

SiO₂ 52.4 - 57.85wt% CaO 22.2 - 39.4wt% MgO 1.96 - 17.4wt% P₂O₅ 0.82 - 7.8wt% B₂O₃ 0 - 1.95wt% Al₂O₃ <1wt%

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A. CLASSI	IFICATION OF SUBJECT MATTER C03C13/00		
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According t	o International Patent Classification (IPC) or to both national	classification and IPC	
	SEARCHED		
Minimum d IPC 6	locumentation searched (classification system followed by class C03C	sification symbols)	
Documenta	tion searched other than minimum documentation to the extent	that such documents are included	in the fields searched
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Date of the	actual completion of the international search	Date of mailing of the	international search report
2	2 September 1997	3 0.	09. 97
Name and	mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk	Authorized officer	
	Tel. (+31-70) 340-2040, Tx. 31 651 epo ni, Fax: (+31-70) 340-3016	Van Bomme	el, L

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